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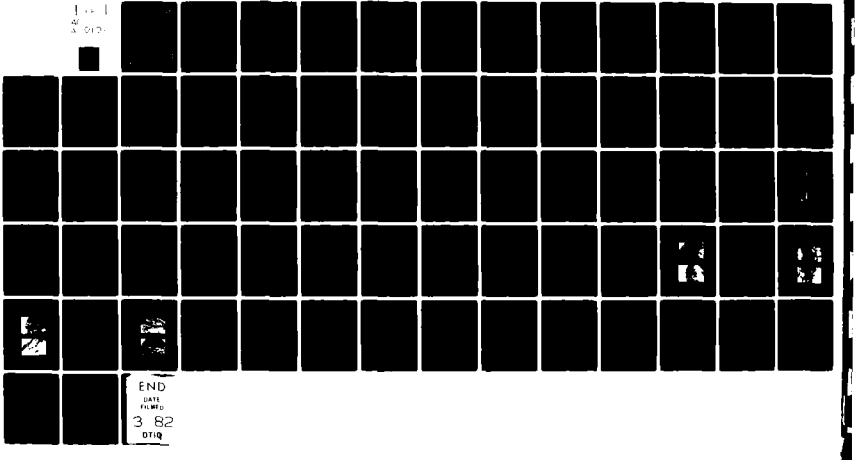

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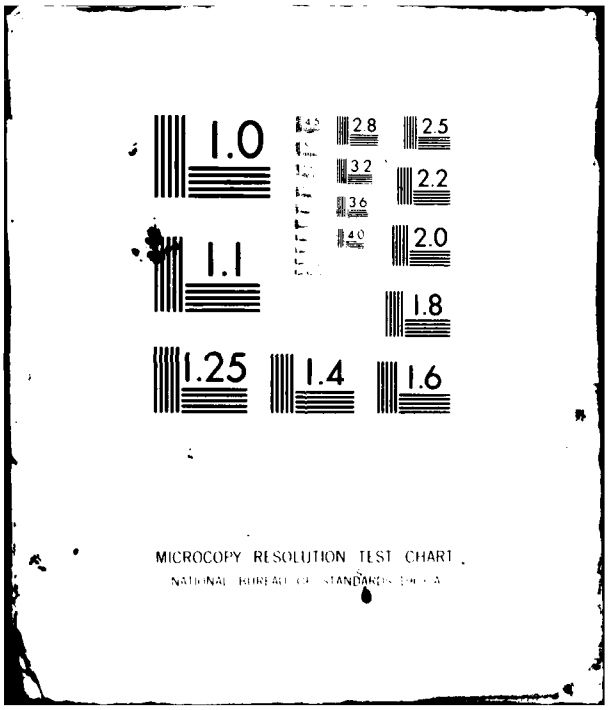
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NATIONAL BUREAU OF STANDARDS-1963-A

# COMPARATIVE TENSILE STRENGTHS OF NON-PRECIOUS DENTAL ALLOY SOLDERS

A

THESIS

Presented to the Faculty of

The University of Texas Graduate School of Biomedical Sciences

at San Antonio

in Partial Fulfillment

of the Requirements

for the Degree of

MASTER OF SCIENCE

by

William Gregory Kaylakie, D.M.D.

San Antonio, Texas

December 1981

COMPARATIVE TENSILE STRENGTHS OF NON-PRECIOUS DENTAL ALLOY SOLDERS

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23 Nov. 1981
Date

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# COMPARATIVE TENSILE STRENGTHS OF NON-PRECIOUS DENTAL ALLOY SOLDERS

Publication No. _____

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The dental profession is ever increasingly faced with the reality of materials' cost, especially precious metal alloys. In recent years, much emphasis has been placed on alternative materials. This heightened attention has resulted in the formulation, improvement and wide spread usage of what is referred to as the base or non-precious alloys. Most of these are chromium-nickel alloys with various other metals added in small varying percentages depending on the manufacturer.

The purpose of this study was to investigate the ultimate tensile strenths of six commercially available non-precious dental alloys and their solders. The alloys and solders tested were: Biobond, Ceramalloy, Cospan, Litecast B, Microbond and Unibond.

Uniform tensile specimens were cast using each alloy. These specimens were then positioned on an aligning device at a gap distance of .33 mm. They were invested and soldered with the high fusing solders supplied for pre-porcelain soldering, using a gas/oxygen torch. Two alloys, Microbond and Biobond, were also tested in the post-porcelain solder mode. All procedures were performed according to the manufacturers' recommendations.

The soldered specimens were all labeled and trued to a uniform diameter on a machinist's lathe. All specimens were subsequently radiographed to determine if any internal defects were present in the solder joint or parent metal. Specimens with apparent radiographic or surface defects were rejected from the final sample group.

Tensile testing was accomplished on an Instron universal testing machine. Ultimate tensile strengths were reported as Mega Pascals and pounds per square inch for comparison with previous studies.

A total of 81 specimen pairs were soldered for all sample groups; of these, 53 were selected for the final sample group. Forty-two of these 53 samples had ultimate tensile strengths greater than those reported for conventional gold solder joints or greater than 45,000 psi.

A one-way analysis of variance showed significant interaction among the pre-solder groups. A Tukey's HSD test showed that Cospan and Litecast B yielded significantly lower values than Microbond, Biobond, Unibond and Ceramalloy pre-solders ($p<.05$).

A two-way analysis of variance and Tukey's HSD showed: Microbond post-solder yielded lower ultimate tensile values than its own pre-solder and Biobond post-solder. Biobond pre- and post-solders did not differ significantly ($p<.05$).

This study found that mean ultimate tensile strength values ranged from 27,473 psi to 79,744 psi for the alloys tested.

Generally, the oven soldered samples showed the least amount of defective and/or broken samples. Future investigation of oven soldering techniques would seem to be indicated for both pre-soldering and post-soldering methods.

## TABLE OF CONTENTS

## LIST OF TABLES

## LIST OF FIGURES

## LIST OF PLATES

# I. INTRODUCTION

The dental profession is ever increasingly faced with the reality of materials' cost, especially precious metal alloys. In recent years, much emphasis has been placed on alternative materials. This heightened attention has resulted in the formulation, improvement and wide spread usage of what is referred to as the base or non-precious alloys. Most of these are chromium-nickel alloys with various other metals added in small varying percentages depending on the manufacturer.

Alloys such as these are used in dentistry to cast crowns and fixed partial dentures to fine degrees of tolerance and accuracy.

The performance criteria for these alloys are such that they are required to:

1) Cast accurately from a wax pattern.
2) Have wear characteristics similar to tooth structure.
3) Be able to be finished in the mouth (burnishable).
4) Chemically resist oral fluids (corrosion and solubility).
5) Have a thermal expansion curve compatable with that of dental porcelains.
6) Be able to be soldered, assembled and resist fracture under masticatory forces.
7) Be biocompatable with the oral tissue (host acceptance).

Most casting and soldering techniques used today in dentistry are those which were developed specifically for dental gold alloys. The newer, non-precious alloys have higher melting ranges and various other characteristics unlike the familiar gold alloys.

The purpose of this study is to investigate the solder bond strength of these non-precious alloys by tensile testing of uniform samples.

Cast crowns are assembled and soldered either before or after the application of dental porcelain; therefore, where ever possible, the solders will be tested for both techniques, pre-solder and post-solder.

Practical application and routine use of these alloys has drawn varied and often conflicting reports from clinicians and laboratories concerning the alloys' ability to be cast and soldered consistently.

Of primary importance are the questions:

1) Is the solder joint capable of withstanding the forces of mastication?
2) Can the soldering procedures be followed and yield consistently acceptable results?
3) Where will the failures or fractures occur (solder, parent metal or interface of the two)?
4) What, if anything, might be done to improve or avoid structural failures of the solder joint?

Since soldering cast units of these alloys has been done rather empirically and often with poor results, this study will objectively address and evaluate these problems.

## II. LITERATURE REVIEW

Simply stated, soldering is the joining of metals by the fusion of intermediary alloys which are of a lower melting point.[1]

Historically, soldering involves the joining of metals with alloys which fuse below 800°F. Brazing is the joining of metals with a low fusing alloy which has a melting temperature greater than 800°F. Welding is the procedure of actually melting or fusing the metals together without the use of an intermediary alloy.

One of the first attempts to evaluate dental solder strengths was by Coleman (1928).[2] He listed some components and ultimate tensile strengths of the handful of gold solders available at the time.

Skinner and Phillips wrote, "A well formed solder joint is one in which the solder wets the cast surface and creates adhesion by primary metallic bonding with the interface between the metal and the solder clearly visible."[1]

Concerning soldering non-precious to precious alloys, Walters (1976)[3] wrote of one clinical situation. He reported using a non-precious metal pontic soldered to a Type III gold abutment crown with #650 fine gold solder. Microscopically, the non-precious metal appeared not to bind at the interface with the gold solder. These observations were, however, not followed by any stress tests or further analysis. It may only be implied here that the higher fusing non-precious alloy did not sufficiently join the solder to its surface, thereby forming a weak solder joint.

Sloan et al. (1980)[4] reported soldering various combinations of alloys with their recommended solders, using precious, semi-precious and non-precious metals. They stated that when base alloys were involved, the resultant fracture was most often

at the interface of the solder and the parent metal. Gold solder joints, however, most often fractured within the solder itself.

A. Gap Distance

Traditionally, the distance between the cast units to be soldered has been limited by the mechanical means of separation, for example, by the thickness of rotary separating disks or by other means of joint preparation.

There are various findings in the literature regarding gap distance. Ryge[5] states that the strength of the joint is dependent on the distance between the units to be soldered and recommends the gaps be greater than .002 - .004 inches to allow for thermal expansion of the units being soldered.

In testing pre and post-ceramic solder joints, Stade et al. (1975)[6] found wider gaps, up to 1 mm., produced stronger joints in Type III gold solders. Rasmussen (1977)[7] reported basically the same results but discovered that with non-precious metals, there were no statistically significant strength differences for varying gap distances. Both studies reported that very narrow gaps of less than .13 mm. were unpredictable and often produced incomplete unions.

Kava et al. (1980)[8] reported that when using gold solders, the authors measured an overall thermal expansion of .003 inches, thus corroborating the findings of earlier investigators. Shear strength of the joints was reported to be relatively constant. Values ranged from 40,000 to 45,000 psi, independent of gap distances up to 1 mm.

B. Alternative Methods of Soldering

Dental soldering procedures have historically caused difficulties and failures for the clinician.

In reference to structural defects, Mumford and Ridge (1971)[9] stated that most failures of fixed partial dentures were the result of solder joint breaks. To improve

this situation, they proposed sectioning the pontic midway and soldering this entire surface rather than the conventional contact area between the two crowns. They reported very good clinical success with this method.

Winslow (1955)[10] proposed leaving 10 to 15 mm. long #8 gauge casting sprues on the lingual side of the castings. These are to be spot welded together on the cast and then the joints are soldered together to avoid the use of indices and investment. After soldering the units, the sprues were sectioned off.

Soldering without investment was also suggested by Perdigon and Von Eepoel (1956)[11] by using projecting buttons on the proximal surfaces of the abutments to hold the pontic in place for soldering.

The most frequently suggested alternative method has been the solid cast or continuous units. This procedure is said to be easier because there are less steps involved and therefore fewer chances for error.

Many clinicians feel that with the cast joint, the final fit cannot be exact because of movement of the biological structures as well as dimensional changes inherent in the laboratory procedures.

Mumford and Ridge (1971)[9] found cast joints to be of a superior fit. Fusayama et al. (1964)[12] reported using a copper alloy to cast three unit fixed partial dentures. They found the solid castings to be more accurate and that the soldered samples had an overall decrease in length. However, Stackhouse (1967)[13] found just the opposite concerning length change using Type III dental gold specimens. He reported an overall increase in length after soldering.

Laser welding has been reported by Gordon and Smith (1972)[14] and Preston and Reisbick (1975)[15]. This technique has the advantages of being able to assemble the units on the cast without indexing, investing or heating. The finished joint also

yields high strength values. The disadvantages are that since no additional metal is added, care must be taken not to perforate the crowns. The welds form a series of sharp overlaps which are very difficult to finish smoothly. Paramount in the consideration of using this technique is the overwhelming cost of the sophisticated equipment.

C. Testing and Evaluation of the Solder Joint

To date, there are only a handful of reported experiments which actually test the strength of the dental solder joint.[2,4,6,7,8,16,17] Most of these studies were primarily concerned with the gold or precious solders.

In one study, Bogan[16] evaluated four different gold solders, all with Type III dental gold. Small cylinders were cast and then soldered using varying gap distances from 0.00 mm. to 0.52 mm. These samples were then subjected to a three point flexure test to fracture. Solder joint fractures were reported under loads varying from 122.5 to 195.5 lbs. with a 2 mm. cross section.

Rasmussen,[8] Stade[7] and Sloan[4] each independently tested gold, semi-precious and non-precious solders. Their findings were all similar and were reported as ultimate tensile strengths ranging from 30,000 psi to 87,000 psi for the non-precious alloys and their corresponding solders.

Non-destructive flexure testing of solder joints was reported by Wictorin (1972).[17] Mild pressure was placed on the joints as they were irradiated by a monochromatic, coherent laser beam (holography). In the analysis of the resultant patterns, any deformation appeared as overlapping lines. They found that internal defects were detectable by this analysis.

Hendrickson (1973)[18] reported a radiographic technique for the evaluation of dental solder joints to increase clinical reliability of these restorations.

Radiographic analysis of dental castings to disclose internal defects were also described by Mattila (1964)[19] and Pascoe and Wimmer (1978)[20]. These methods all involved industrial radiographic equipment of high KVP ratings, on the order of 150 to 200 KVP. Such equipment, of course, is not available in the dental office; dental units produce only up to 90 KVP.

Wise and Kaiser (1979)[21] described a technique for radiographic examination of non-precious metal castings using standard dental x-ray equipment. All of the aforementioned studies reported finding defects radiographically which were not apparent on visual examination of their sample castings. The reported percentage of dental castings sampled which contained apparent internal defects ranged as high as 79% (Mattila, 1964).[19]

D. Intraoral Forces

The most logical question regarding all of the aforementioned data is: What does it mean clinically and how does this compare with intraoral forces?

Klaffenbach (1936)[22] used one of the first scientific approaches to biting force analysis. He measured biting force on a large sampling of patients with an intraoral device. He reported biting forces from 45 to 295 lbs. in various subjects. This data was transferred into units of stress per area and he extrapolated pressures of 117 to 10,416 psi which might be found where a cusp tip occludes against an opposing fossa or marginal ridge.

Brumfield (1954)[23] proposed an elaborate sequence of proportions to calculate the stress placed on a gold "dental bridge." Utilizing direct laboratory measurements, he considered span length, width and height. He reported that the working stress for a typical gold bridge is approximately 27,000 psi or 60% of the proportional limit of a Type III gold solder joint of 45,000 psi.

Using a transducer, DeBoever et al. (1978)[24] reported occlusal forces in posterior pontics of 2.03 to 4.26 lbs. Only a few of their readings even exceeded 15 lbs.

Most recently, Gibbs et al. (1981)[25] reported using sinusoidal sound vibration introduced at the forehead with a piezoelectric crystal transducer used in conjunction with electromyography. This study eliminated the need for intraoral devices. Biting forces ranged as high as 66.5 lbs. on swallowing and 58.7 lbs. during chewing. They also reported a maximum clenching force range of 55 to 280 lbs.

E. Physical Properties of the Alloys

Information concerning the physical properties of the non-precious alloys are of importance.

These dental casting alloys are mostly all variations of a basic chromium-nickel alloy[26,27]. Each manufacturer has his own formula and process which involves minor elemental changes and proportional variations.

Minor elemental changes can enhance or mitigate certain physical properties. Rowe et al. (1974)[28] experimented with a cobalt-chromium-nickel non-precious alloy in a 30-40-30 proportion. The composition was varied by adding small amounts of tantalum to increase the strength. This modifier did in fact increase the strength when added in small (1%) increments until it comprised 6% of the total alloy. However, it also decreased the ductility in a proportional manner.

Although many of the modifying elements are not always stated by the manufacturer, it is known that some of these alloys contain 1-2% beryllium. The purpose of this element is to inhibit excess oxide formation which in turn prohibits adequate porcelain bonding. Difficulties in controlling the oxide layer were clearly stated by Sced and McLean (1972)[29] when they described porcelain fractures from

the surface of these metals. Beryllium, as well as silicon, additions are also reported to facilitate the flow of molten metal upon casting.[30]

Regarding the microstructure, Lewis (1979)[31] investigated the microsegregation on solidification of two non-precious dental alloys. He found primary crystallized dendrites of the nickel-chromium or cobalt-chromium matrix solid solutions which were subsequently surrounded by interdendriticly formed eutectic carbides.

When these alloys are cast, certain procedures must be followed which differ from the conventional precious gold alloys. Jendresen (1975)[32] recommended using burnout temperatures of 1500-1700°F. to maintain mold temperatures which allow sufficient expansion of the phosphate-bonded investment for casting accuracy.

Regarding casting of non-precious alloys, Cole and Vincent (1980)[33] reported that casting two non-precious alloys in standard metal casting rings produced undersized castings. They recommended wax paper casting rings to allow for proper expansion of the casting investment; this procedure yielded a more accurate finished casting.

## III. MATERIALS AND METHODS

Dental materials research is carried out primarily to evaluate the available proprietary materials in a manner closely approximating actual clinical and laboratory use.

There are many variables involved with handling most materials of which the operator should be aware. An error or oversite in technique could, in many instances, cause failure of the procedure. In order to make an accurate comparison, it is therefore best to standardize the technique whenever possible.

Testing of the solder joints in the tensile mode was chosen for two reasons:

1) Transverse or three point flexure testing was first considered because it more closely resembles clinical use, but the internal stress is more complex in nature and therefore may have more variables.

2) Although fatigue failure is most probably the failure mechanism for the majority of prosthetic appliances under occlusal function, it could not be within the scope of this study to determine fatigue strengths of all these solder bonds. Since the majority of previous literature has documented solder bond strengths as ultimate tensile strengths, it was decided to also use this technique for comparison purposes. In addition, the tensile test is known to be a quick determination and a valuable tool for numerous measurements.

The tensile testing mode was chosen to standardize the methods and data to be obtained in this study.

A. Test Specimens

The cast alloy specimens to be used were designed according to the American Society of Testing Materials specification for tension testing of metallic materials.[34]

Individual specimens to be soldered together were 12 mm in length with a diameter in the area to be joined of 2.5 mm (Figure 1).

The total length of the soldered specimens was therefore about 24 mm. A master stainless steel die one-half of the finished tensile test bar was machined to specifications. Molds of this die were then made in a silicon impression material (Vescote, Teledyne Products, Elk Grove Park, Illinois). These molds were then injected with melted inlay wax (Maves Co., Cleveland, Ohio) and allowed to cool to room temperature for 30 minutes.

The wax patterns were then carefully separated and inspected for voids.

Acceptable wax specimens were then sprued onto a continuous number eight gauge wax sprue bar (Sybron/Kerr Manufacturing, Romulus, Michigan) with a continuous flat feed sprue of standard pink base plate wax.

The sprued specimens were then invested in High Temperature phosphate bonded investment (Whip-Mix Corporation, Louisville, Kentucky). A double layer of asbestos was used in each ring to allow for adequate thermal expansion to insure accurate specimen size (Figure 2).

All invested specimens were allowed to bench set for 60 minutes. They were then placed into a cool burnout oven and the temperature was allowed to rise to 1550 degrees Fahrenheit. Upon reaching this temperature, they were held there for 60 minutes before casting.

Figure 1. Diagramatic Representation of One Cast Metal Specimen.

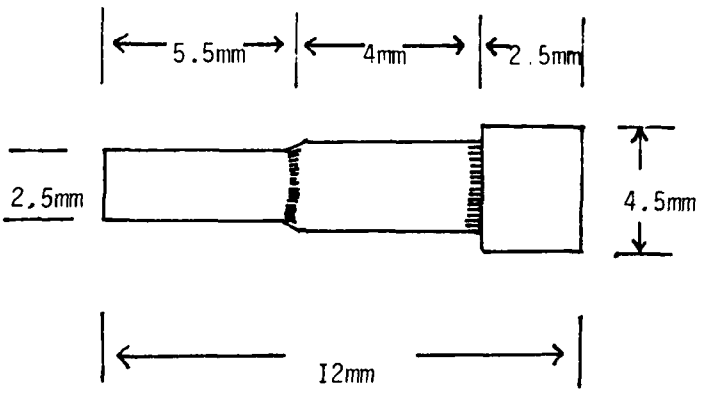
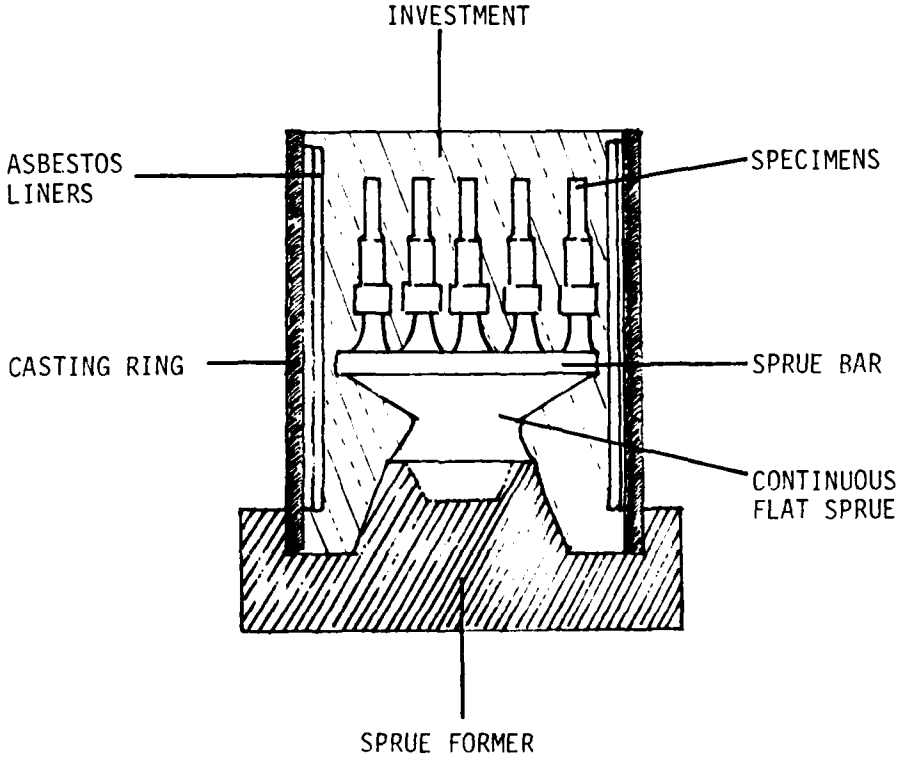

5.5mm
4mm
2.5mm
2.5mm
4.5mm
12mm

Figure 2. Cut Away Diagram of Casting Ring Showing the Position of the Specimen Patterns in the Ring Before Casting.


INVESTMENT
ASBESTOS LINERS
SPECIMENS
CASTING RING
SPRUE BAR
CONTINUOUS FLAT SPRUE
SPRUE FORMER

All casting was done on a centrifugal broken arm dental casting machine. The alloys were melted with a gas/oxygen flame using a multioriface tip as recommended by the manufacturer (KL-R Perplex, Perkeo Company, West Germany).

Because these non-precious metals have approximately one-half the specific gravity of conventional dental alloys, greater amounts were used to ensure complete casting of all the samples.

Each manufacturer supplies his metal in various ingot sizes. Generally, five to eight ingots of alloy, approximately 33 gms, were used to cast each ten specimens.

After casting, the rings were allowed to cool to room temperature. The casting was then removed from the ring by gentle tapping and the remaining phosphate bonded investment was removed by sand blasting.

All individual specimens were then separated from the casting sprue using a #225 carborundum separating disk (Ticonium Company, Albany, New York), on a high speed lathe.

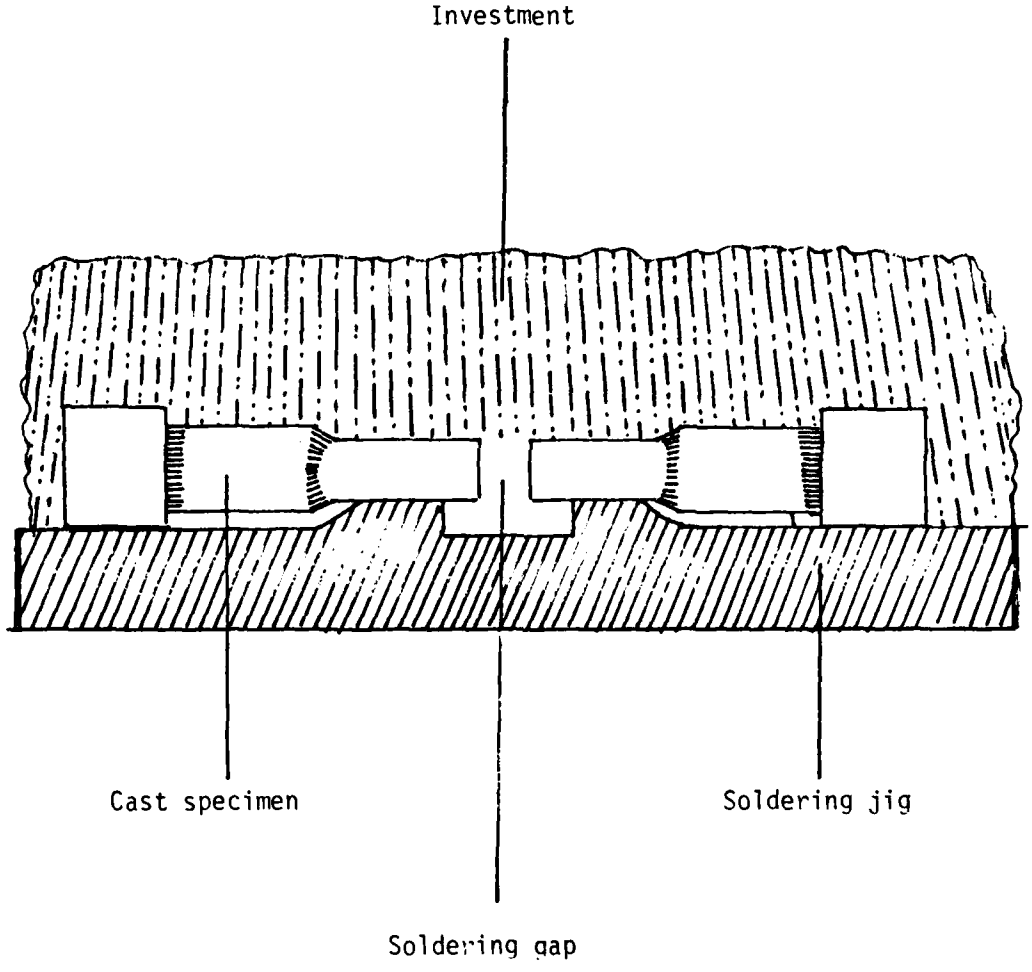
The specimens were then examined under magnification and any casting nodules were lightly removed with #205W aluminum oxide stones (Ticonium Company, Albany, New York) mounted in a laboratory handpiece. The ends to be joined were also prepared with this stone.

At this point, the specimens were measured with a Vernier caliper to assure that they were accurate to within .01 mm of the master stainless steel die.

B. Investing for Soldering

A soldering holding jig was machined from stainless steel so five pairs of specimens could be aligned coaxially with a variable gap distance (Figure 3). A gap distance of .33 mm was chosen because this dimension was within the range

Figure 3. Diagram of Two Invested Cast Specimens in Position on the Soldering Jig.


Investment
Cast specimen
Soldering jig
Soldering gap

recommended by all of the manufacturers and previous studies had shown this to be a favorable separation for dental soldering.

The specimens were tacked to the soldering jig with Sticky Wax (Syborn/Kerr, Emeryville, California) while a metal feeler gauge was held between the prepared ends to ensure even separation.

High heat soldering investment was mixed according to the manufacturer's directions (Whip-Mix Corporation, Louisville, Kentucky).

Hand spatulation was used to mix the investment in a rubber bowl using 100 grams of powder to 28 ml of distilled water. The investment was gently teased over each pair of specimens and stacked with a plaster spatula. The investment was allowed to set for one hour. After this tme, the invested specimen pairs were lifted from the jig and each pair was trimmed into a rectangular block (Figure 4). The investment was trimmed away from each side of the joint in a conical fashion to allow access for the soldering flame.

The invested samples were then flushed with boiling water to remove all wax and debris from the blocks.

From this point, there were slight variations in the handling of each alloy as specified by the manufacturers.

C. Pre-Soldering

Drying of the investment blocks was similar for all of the alloys, but the temperatures and holding times differed slightly (Table 1). Other varying factors such as oxygen pressure to the torch, solder form as supplied and time the torch flame remained in contact with the joint after the solder flowed are also listed.

All pre-soldering was accomplished with a natural gas/oxygen torch, Perkeo #65 Torch and Perkeo P-2-4 Tip (Perkeo Manufacturing Company, West Germany).

Figure 4. Diagram of Invested Specimens as Pre-Soldered on Soldering Stand. Note the Sliceway Cut into the Investment to Allow Adequate Heating of the Joint by the Flame.

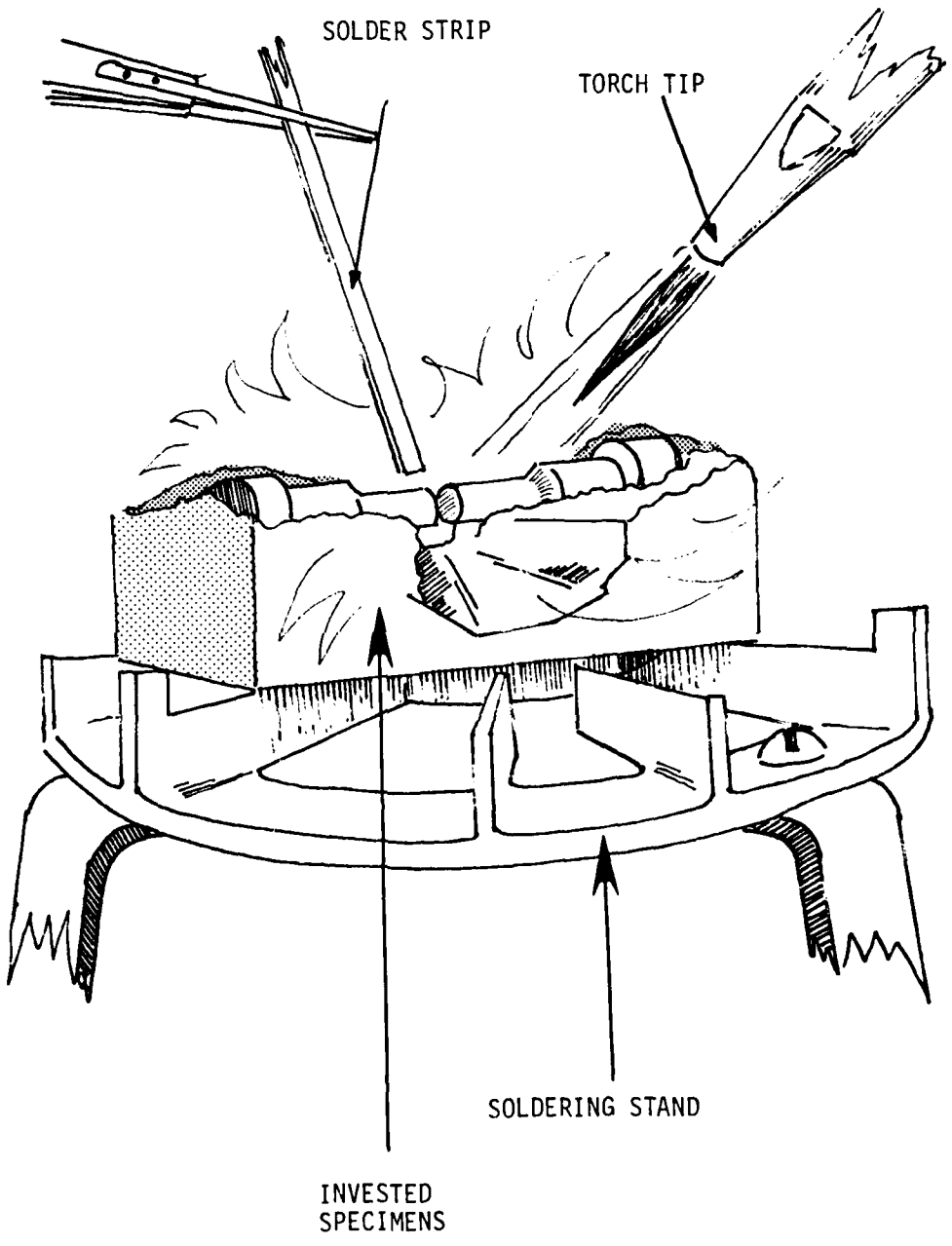

SOLDER STRIP
TORCH TIP
SOLDERING STAND
INVESTED
SPECIMENS

**Table 1.** TECHNIQUE VARIATIONS AMONG PROPRIETARY NON-PRECIOUS ALLOY PRE-SOLDERS TESTED.

| Alloy | Solder Form | Investment Drying Temp. | Oxygen Pressure | Flame Contact After Solder Flows |
|---|---|---|---|---|
| Biobond | Strip | cold furnace to 1400°F | 5 lbs. | None |
| Ceramalloy | Rod | 1200-1500°F | 3 lbs. | None |
| Cospan | Rod | 1100°F | 6 lbs. | 60 sec. |
| Litecast B | Strip | 1200°F | 5 lbs. | 5 sec. |
| Microbond $NP_2$ | Strip | heat only enough to dry investment | not specified | None |
| Unibond | Paste | at mouth of 900°F furnace | 3 lbs. | None |

The flame was adjusted so that the reducing portion of the flame was three to six inches from the tip of the torch, depending on the specified oxygen pressure.

Before any specimens were soldered, each alloy and its corresponding solder were trial tested on prepared and fluxed areas of the original sprue pieces. This was done to become more familiar with the handling qualities of each solder.

The specimen blocks were removed from the oven. A small amount of the supplied flux was placed into each gap if the specimens were not prefluxed. The blocks were placed on a soldering stand, and the flame was applied to the joint keeping the reducing zone over the soldering area. Welder's goggles were worn for eye protection. When the specimens glowed red-orange through the glasses, the solder was touched to the gap area until it flowed into and filled the gap. The solder rod or strip was immediately withdrawn and the flame was held for the specified time or withdrawn, according to the manufacturer's instructions. One alloy and solder was supplied in a paste form which had the flux and solder granules in a suspension. This was applied before drying the investment. The flame was applied in the usual manner until the granules melted and fused.

D. Post-Soldering

Post-soldering was tested on only two of the alloys used in this study. This technique is used clinically after porcelain has been fused to the metal and has been final glazed. The post-solder therefore must melt below the fusing temperature of porcelain. If a direct flame is placed on the porcelain, it can cause porcelain fracture; to avoid this damage, the operation is performed in an oven.

To simulate any possible heat conditioning affects on the alloys, all post-solder specimens were subjected to cyclic firing in a porcelain oven. This simulated the thermal treatment the alloys would receive in a clinical situation. The specimens

were placed in a porcelain oven at 1360°F and raised to 1760°F at a rate of 100°F/minute. They were cooled to room temperature and the cycle repeated four more times to simulate two opaque, two body and one glazing firings.

The specimens were then joined with sticky wax, invested and cleaned in the same manner as the pre-solder specimens.

From this point, both post-solders were handled differently.

1. Biobond post-solder

Biobond C & B soldering flux (Dentsply International Inc., York, Pennsylvania) was applied to the solder joint area. A strip of solder approximately 1/4 inch long was cut and placed in the solder joint area. The invested block was placed in the drying position of the oven for 15 minutes while the temperature was held at 900°F. A small carbon purge block was placed on the floor of the oven *before closing*. A vacuum was applied and the temperature was raised to 1550°F and held for one minute. The vacuum was released; the soldered specimens were removed from the oven and allowed to bench cool to room temperature.

2. Microbond post-solder

Microbond Post-Porcelain Soldering Flux (Howmedica Inc., Chicago, Illinois) was applied to the joint with a clean sabel brush. The invested specimens and flux were dried at the opening of the porcelain oven for 15 minutes while the temperature was held at 900°F. Next, the specimens were placed in the oven and the temperature was raised to 1650°F and held at that temperature for one minute.

A strip of Microbond post solder was dipped into the flux and dried to a glaze over a bunsen burner. The oven door was opened and the tip of the prefluxed solder was touched to the joint area. As the hot metal melted the solder, the strip

was fed in until the joint space filled and then withdrawn. The soldered specimens were removed from the oven and allowed to bench cool.

E. Machining of Specimens

From this point on both pre-solder and post-solder specimens were handed in the same manner.

Each specimen was numbered in the order in which it was soldered. All specimens were sand blasted to remove residual investment and flux. Rouge dissolved in chloroform was used to number each end of each specimen. This was followed by arbitrarily assigning and painting the non-testing area of each specimen in each alloy group with an acrylic color for identification. This was done to avoid any mixing of sample groups during machining and testing.

The specimens were then all trued on a machinist's lathe to an even diameter thickness at the solder joint area. To assure concentricity and accurate diameters for all specimens, they were machined to uniform diameters of 2.05 mm .05 mm.

After completion of the machining, each specimen was again measured to .001 mm at the solder joint area with a vernier caliper.

F. Metal Radiography

As previously discussed, solder joints and castings may contain voids and imperfections not visible upon external examinaton. Therefore, radiographic examination was used to detect any possible internal defects in the solder or parent metals.

A standard dental radiographic unit was utilized (General Electric Corporation, Milwaukee, Wisconsin). In numerical order, the samples were secured on sheets of pink baseplate wax. Each film was labeled with radiopaque symbols for identification and orientation.

The samples were placed on top of a standard intra-oral occlusal film, 2-1/4 inches by 3 inches (Kodak Manufacturing Company, Rochester, New York). The end of the x-ray cone was placed 3 inches from the specimens. A setting of 30 impulses, 15 milliamperes, at 90 Kilovoltage power was used for each exposure. All radiographs were developed in an automatic processor (Phillips Dental Systems, Stamford, CT) utilizing a digital control monitor module.

Any samples with apparent surface defects or internal radiographic defects were recorded for elimination from the final sample group. All samples were, however, subjected to tensile testing to confirm radiographic findings.

G. Tensile Testing

Tensile testing was performed on an Instron Universal Testing Machine, Model 1125 (Instron Corporation, Canton, Massachusetts).

Special slotted holding devices were machined from block steel and heat hardened (Plate 1). These were used to engage and firmly hold the test specimens during testing. The upper crossmember of the machine contained the load cell. The upper holding device was connected to the load cell by a universal joint to avoid torsional forces on the specimens during loading.

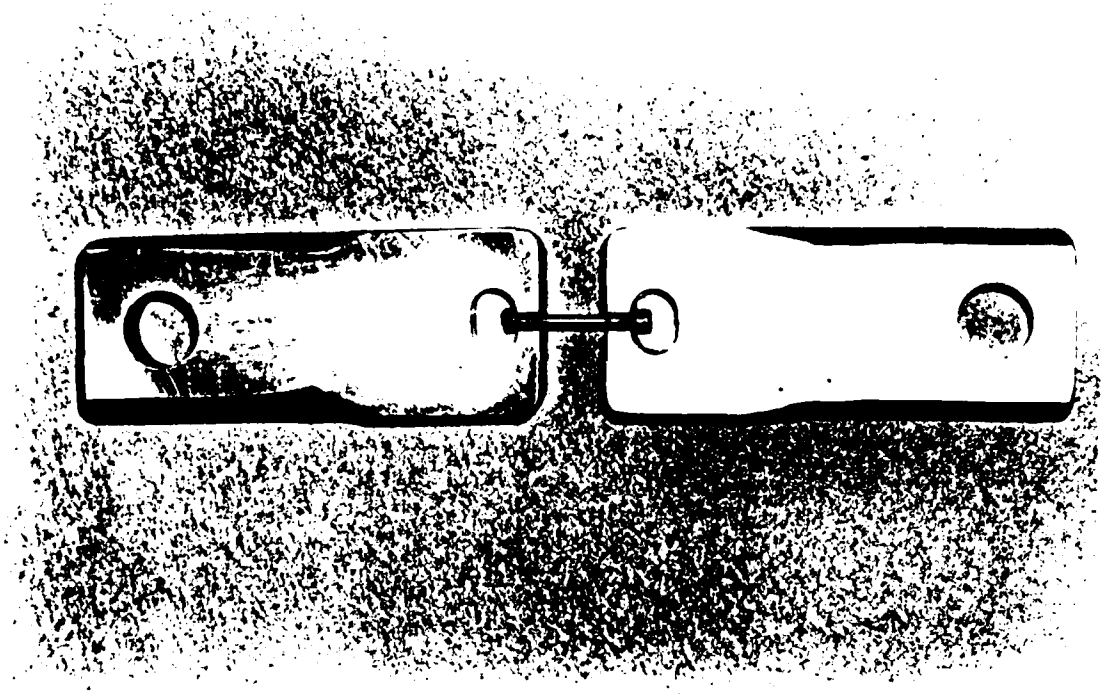
The specimens were inserted into the machined slots and testing was completed to specimen fracture (Figure 6).

The following settings were used on the Instron machine:

PLATE 1

Soldered and Machined Tensile Specimen Positioned in Holding Device

Overall length of this assembly as seen is about (7 inches).



Load cell .....5 Kilonewtons
Full scale load .....1, 2 or 5 Kilonewtons
Chart speed .....50 mm/min.
Crosshead speed .....1 mm/min.

The ultimate tensile strength of each solder joint was recorded on the data sheets as read from the graph recorder chart on the Instron.

Finally, all fractured specimens were viewed under a metallographic microscope (Zeiss, West Germany) to observe the morphology of the fracture surface and to discern whether the fracture had occurred in the solder, the parent metal or at the interface of the two.

Typical fracture surfaces and morphologies were photographed through the metallographic microscope.

H. Statistical Analysis

A consultation was held with the Department of Computer Resources to determine what type of analysis should be given to the data obtained in this study.

Since the primary aim of this study was to compare non-precious pre-solders, it was determined to apply a one-way analysis of variance to the six pre-solder groups. A level of confidence was to be sought at the 95% confidence interval or $p<.05$.

The post-solder groups were compared to their own proprietary pre-solders as well as to each other by a two-way analysis of variance. A level of significance was also determined to be accepted at $p<.05$.

The level of significance will be determined by applying the data to Tukey's HSD (honesty significant difference) test. This test was chosen because of its high degree of stringency especially for small sample groups.[35]

## IV. RESULTS

This study revealed that there was considerable difficulty obtaining consistent results in pre-soldering non-precious dental alloys. This difficulty appears to be both material and technique related.

A total of 81 pairs of specimens were soldered using six different proprietary pre-solders and two post-solders. Seventeen of these solder joints were fractured during the truing phase, before testing. Eleven samples were rejected because of visible surface or radiographic defects. The remaining 53 samples had ultimate tensile strength values which should theoretically withstand interocclusal forces, as reported by Brumfield (1954).[24] Of these 53 samples, 42 had ultimate tensile strengths exceeding the 45,000 psi reported for conventional gold solder.

A one-way analysis of variance was applied to the pre-solder values. The interaction indicated was further described by a multiple comparison Tukey HSD test showing that Cospan and Litecast B solders were significantly different from Microbond, Biobond, Unibond and Ceramalloy solders at the 95% confidence level ($p < .05$) (Table 2) (Figure 6).

A two-way analysis of variance was applied to the post-solder values along with the pre-solder values of the same proprietary alloys. The results of the subsequent Tukey test showed that there was a significant difference between the values obtained for Biobond post-solder and Microbond post-solder ($p<.01$) (Table 3) (Figure 7). The values obtained from Microbond pre-solder significantly differed from those obtained from Microbond post-solder ($p<.01$). There was no significant difference between Biobond pre-solder and post-solder values.

**Table 2.** ULTIMATE TENSILE STRENGTHS OF ALL SOLDER JOINT VALUES USED IN STATISTICAL ANALYSIS.
MPa (Mega Pascals) = mega newtons per square meter($MN/M^2$)
PSI = pounds per square inch conversion

**PRE-SOLDER**

| BIOBOND | | MICROBOND | | UNIBOND | |
|---|---|---|---|---|---|
| MPa | PSI | MPa | PSI | MPa | PSI |
| 363.9 | 52,772 | 432.7 | 62,735 | 518.8 | 75,225 |
| 450.4 | 65,312 | 479.6 | 69,538 | 490.9 | 71,185 |
| 565.7 | 82,028 | 504.2 | 73,109 | 490.4 | 71,108 |
| 364.3 | 52,819 | 434.7 | 63,043 | 502.5 | 72,856 |
| 573.4 | 83,139 | 490.8 | 71,160 | 624.4 | 90,540 |

| CERAMALLOY | | LITECAST B | | COSPAN | |
|---|---|---|---|---|---|
| MPa | PSI | MPa | PSI | MPa | PSI |
| 377.4 | 54,721 | 135.6 | 19,659 | 330.5 | 47,927 |
| 428.4 | 62,120 | 71.6 | 10,383 | 187.9 | 27,249 |
| 455.6 | 66,057 | 341.7 | 49,557 | 324.4 | 47,032 |
| 499.2 | 72,385 | 204.9 | 29,709 | 106.6 | 15,459 |
| 377.4 | 54,724 | 193.5 | 28,059 | 325.0 | 47,082 |

**POST-SOLDER**

| BIOBOND | | MICROBOND | |
|---|---|---|---|
| MPa | PSI | MPa | PSI |
| 555.7 | 80,578 | 364.9 | 52,911 |
| 509.9 | 73,946 | 325.5 | 47,198 |
| 581.6 | 84,328 | 252.5 | 36,619 |
| 605.4 | 87,779 | 250.8 | 36,366 |
| 497.2 | 72,090 | 184.5 | 26,746 |

Figure 5. Mean Ultimate Tensile Strength Values of Pre-Solder Samples. Vertical line at the top of each bar represents standard deviation.

MB - Microbond
BB - Biobond
UNB - Unibond
CMY - Ceramalloy II
CSP - Cospan
LCB - Litecast B

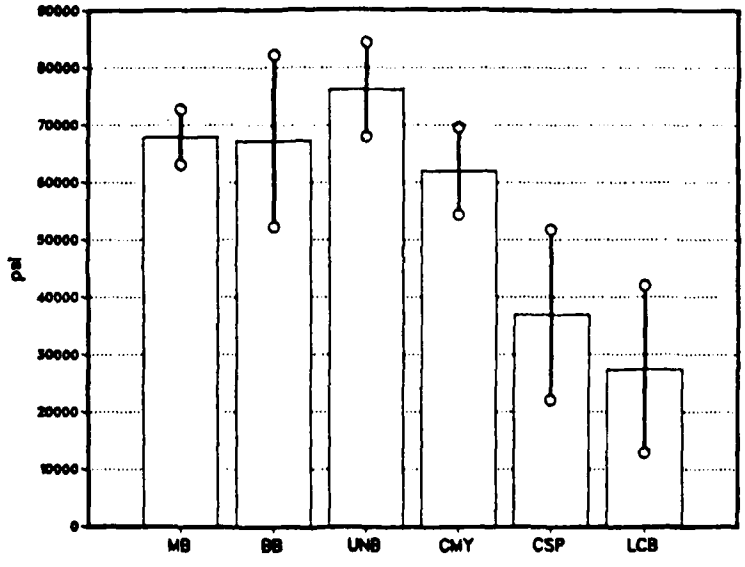

psi
80000
70000
60000
50000
40000
30000
20000
10000
0
MB
BB
UNB
CMY
CSP
LCB

**TABLE 3.** MEANS AND STANDARD DEVIATIONS FOR ULTIMATE TENSILE STRENGTH VALUES.

(All values are in PSI, pounds per square inch)

(Conversion factor to MPa = $\frac{PSI}{145}$)

**PRE-SOLDER**

| | MICROBOND | BIOBOND | UNIBOND |
|---|---|---|---|
| mean | 67,917 | 67,214 | 76,183 |
| std. dev. | 4,762 | 14,937 | 8,198 |

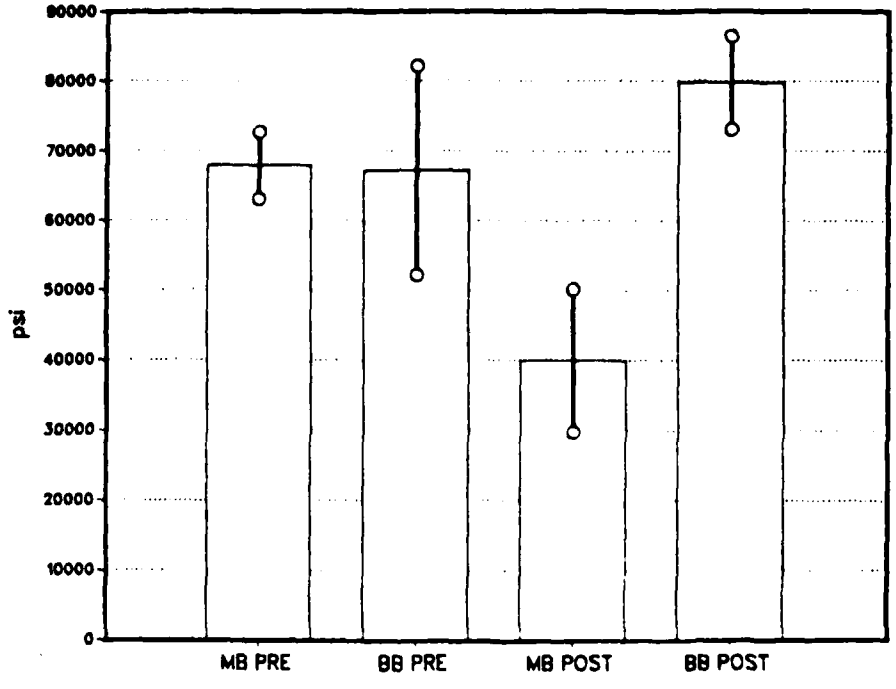
| | CERAMALLOY II | COSPAN | LITECAST B |
|---|---|---|---|
| mean | 62,001 | 36,950 | 27,473 |
| std. dev. | 7,587 | 14,839 | 14,545 |

**POST-SOLDER**

| | MICROBOND | BIOBOND |
|---|---|---|
| mean | 39,968 | 79,744 |
| std. dev. | 10,232 | 6,680 |

Figure 6. Mean Ultimate Tensile Strength Values for Post-Solders and Their Proprietary Pre-Solders. Vertical line at the top of each bar represents standard deviation.

MB PRE - Microbond Pre-Solder
BB PRE - Biobond Pre-Solder
MB POST - Microbond Post-Solder
BB POST - Biobond Post-Solder


90000
80000
70000
60000
50000
40000
30000
20000
10000
0
psi
MB PRE
BB PRE
MB POST
BB POST

All of the solders tested yielded some solder joints with strengths exceeding those of conventional gold solder joints; however, some alloys had a large number of low strength joints. Difficulties were encountered soldering each of these non-precious alloys by the gas-oxygen torch method.

Regarding the number of broken or eliminated samples prior to testing, among all of the pre-solder samples, the most consistent results were obtained with Unibond (paste solder), Biobond (strip solder) and Microbond (strip solder). Microbond pre-solder was also statistically the most consistent.

The most consistent solder, regarding the total number of samples able to be tensile tested,, was Biobond post-solder. This alloy was oven soldered under vacuum. This solder had consistently high tensile strength and no incomplete solder unions.

All of the selected samples in this study fractured through the solder itself except for three Microbond post-solder specimens which fractured at the interface of the solder and the parent metal and one of the Microbond pre-solder samples which fractured in the parent metal. Therefore, 94% of the successfully soldered joints fractured through the solder itself.

Radiographic analysis proved to be a valuable means of detecting internal defects, especially in the rod and paste forms of solder.

All of the solders tested exhibited a brittle fracture except for Microbond pre-solder specimens which showed large elongation of both the parent metal and the solder.

Microscopic examination of the fracture surfaces revealed a typical spherical or "wet sand" appearance. There were voids of varying sizes in many of the fractured surfaces. In general, there appeared to be more voids as the joint strength

decreased; some of the weakest joints even had flux inclusions along the fracture surface (Plates 2-5).

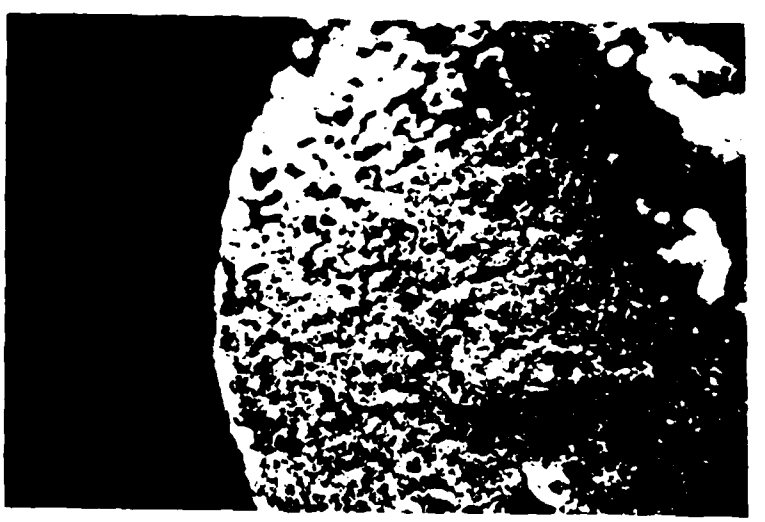
# PLATE 2

Fracture Surface of Biobond Pre-Solder Samples

Magnification as printed (300x)

A. Weakest sample for this group-surface of the solder, however, appears fairly even and granular.

B. Strongest sample for this group. The surface is similar to (A) except that the fracture has occurred in different planes.



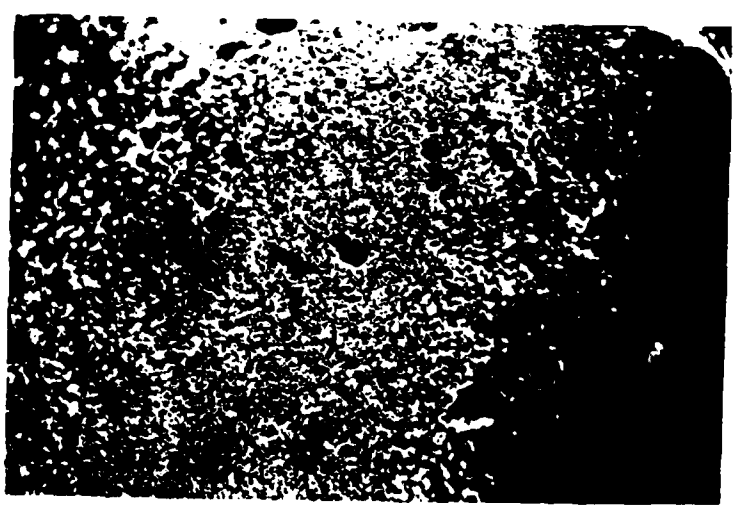
# PLATE 3

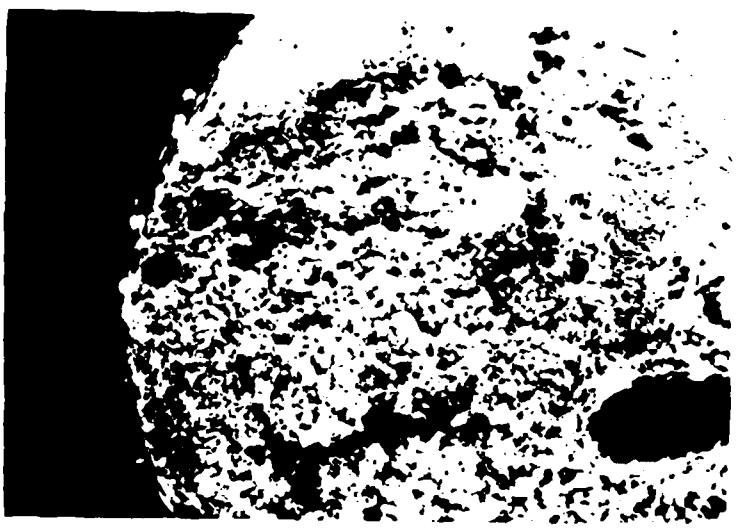
Fracture Surface of Microbond Post-Solder Samples

Magnification as printed (300x)

A. Fracture surface of typical weak solder joint. Some of the parent metal is visible (upper right hand corner).

B. Typical strong solder joint. Fracture is completely through the solder. Numerous small voids appear within the solder.





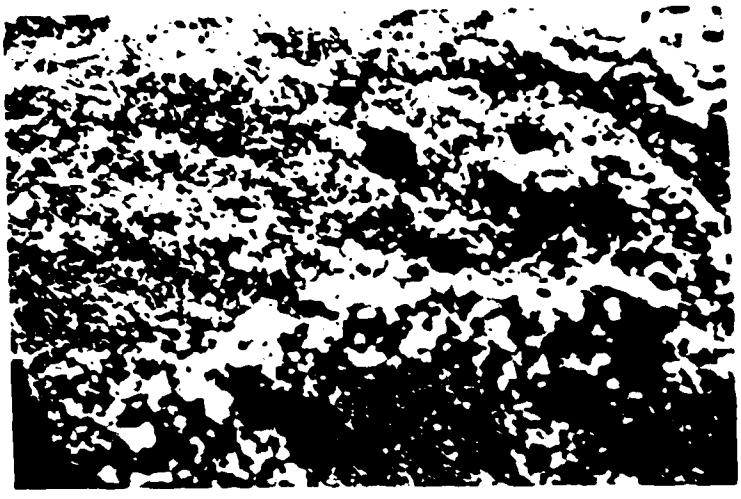
# PLATE 5

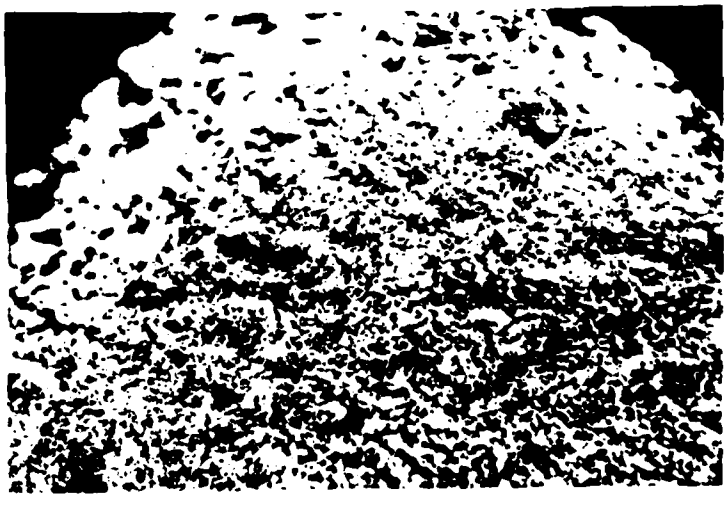
Fracture Surface of Unibond Pre-Solder Samples

Magnification as printed (300x)

A. Weakest sample of this group. The surface is irregular with numerous voids. A few small spherical inclusion appear (upper center of photomicrograph).

B. Strongest sample for this group. Surface appears similar to (A) but there are few is any voids.





## V. DISCUSSION

This study shows that the six commerical alloys and solders could all be successfully soldered; however, there are inconsistencies among the various alloys in obtaining predictable solder unions. Eighty-one soldered specimens were prepared, of these 28 were defective or fractured before testing. This would be a poor success rate for most laboratory procedures. Thus, material problems may be related to techniques, materials and the stresses induced upon the specimens by machining.

The ultimate tensile strength values obtained in this study are similar to those reported by Preston (1980) and Rasmussen (1979); these literature values ranged from 17,000 psi to 87,000 psi.

Sloan (1980)[4] reported most fractures of non-precious solder joints occurred at the interface of the solder and the parent metal. This study, however, found most solder joint breaks (94%) occurred in the solder itself. The reasons for these contradictory findings may be in the technique employed as well as in the proprietary alloys tested.

This study, or any study which evaluates metal castings and solder joints, must consider and account for the possibility of internal defects in the specimens which could adversely affect the final results. Solder joints with obvious or hidden defects can incorrectly influence material evaluation when the defects may in fact be caused by technique difficulties. In this study, all solder joints were radiographed. The relatively short radiographic exposure (30 impulses, 1/2 sec.) successfully penetrated the specimens. It did not penetrate the more dense strip solders. These solders, however, could be clearly seen at the interface of the cast specimens.

The non-precious solders which were supplied in a rod or paste form appeared to have the same amount of radiopacity as the cast specimens. Voids and partially joined interfaces could be seen on the radiographs. The more the solders appeared to blend in or present a homogeneous appearance radiographically, the stronger were the resultant tensile strengths for those joints.

Joints with visible voids or continuity breaks at the interface were rejected from the sample group. All samples, however, were subsequently tensile tested to confirm the radiographic findings.

A number of factors must be considered in analyzing the results of this study. Numerous opportunities exist for induced error in these soldering procedures; these include: operator, environmental, materials and technique.

A major consideration with the non-precious alloys is the control of surface oxide formaiton; this problem has been encountered for years with similar alloys in the aviation industry. Most industries today successfully combat this soldering and welding problem with the use of vacuum or protective atmosphere ovens while dental technology appears to be still relegated to protection and cleaning of solder joints with fluxes. A more directed approach employing vacuum and/or protective gas environments for dental soldering would appear beneficial.

As seen in the results, none of the torch soldered samples yielded as consistent results as those post-soldered specimens which were oven soldered under vacuum.

The natural gas used with the torch may contain some trace contaminants (carbon) as well as a small allowable amount of moisture. One manufacturer (Williams Gold Company) recommended the use of a propane torch rather than natural gas, if possible.

The Microbond post-solder samples were oven soldered also, but without vacuum. They yielded lower ultimate tensile strength values. This may be due in part to the availability of oxygen to the hot specimens as the open oven door technique is employed. It is also very likely that the oven temperature fluctuated as the cool room air mixed with the hot oven air. This possible surface oxide formaton may also explain why these were the only samples which exhibited fractures at the interface.

The Biobond post-solder samples were soldered in the porcelain oven, under vacuum, with a small block of carbon to purge impurities. These specimens appeared to be the most consistent number of samples able to be soldered and tensile tested because of even heating and controlled atmosphere to minimize contaminaton of the solder joint.

The pre-solders which were supplied in rod forms performed with some inconsistencies. The fractured surfaces of these specimens showed inclusions and voids of various sizes, apparently caused by entrapped gases.

Control of surface oxides and contaminants is imperative with these alloys. Some of the specimens of each of the alloys and solders tested were adequately joined; therefore, the variability would seem to rest with the technique or the operator.

The author, feels if these solders are to be used in the future, that oven soldering methods should be investigated to assure more predictable and uniform results for the operators.

The paste solder supplied for Unibond metal was the one non-strip solder that performed very well. This may be because the solder is ground to a powder and mixed with the flux. All the solder is flux coated until it begins to melt and fuse;

this material preparation reduces atmospheric contact and chances for oxidation and contamination.

This study reports ultimate tensile strengths for six commercial non-precious solders. The intent of this study was to determine whether the alloys prepared according to the manufacturers' recommendations could yield ultimate tensile solder strengths which could withstand interocclusal forces.

This study does not, by any means, answer all questions concerning these dental alloys. Additional research in the areas of fatigue testing, accuracy of soldering and interfacial elemental profile analysis of the solder joints needs to be performed.

Fatigue testing is important because it should yield data which is more useful to the clinician who can relate this information to service life of a prosthesis under masticatory forces.

Accuracy testing is needed to assure that soldered units will fit precisely as indexed.

Elemental analysis might help to understand the metallurgical changes occurring in the solder joint and possibly identify some technique or compositional problems.

## VI. SUMMARY

This study investigated the ultimate tensile strengths of six commerically avaialble non-precious dental solders. The alloys and their supplied solders tested were: Biobond, Ceramalloy II, Cospan, Litecast B, Microbond and Unibond.

Uniform tensile specimens were cast for each alloy. The pre-solder specimens were all soldered using a gas-oxygen torch as recommended by the manufacturers. Two of the alloys, Microbond and Biobond were also post-soldered in a porcelain oven following the manufacturers' recommended methods.

Some difficulty was encountered obtaining consistent solder unions, especially with the gas-oxygen torch method.

Eighty-one specimen pairs were soldered; fifty-three of these were deemed acceptable for tensile testing after machining and radiographically inspecting the solder joints.

Statistical analysis showed:

1. Microbond, Biobond, Unibond and Ceramalloy II pre-solders were not statistically different from each other, but were statistically different from Litecast B and Cospan which showed significantly lower ultimate tensile strength values.
2. Biobond post-solder was significantly stronger than Microbond post-solder.
3. Microbond pre-solder was significantly stronger than its own post-solder.
4. Biobond pre-solder and post-solder did not differ significantly.

Since soldering of these non-precious alloys yielded some inconsistent results, further investigation is necessary in oven soldering, metallographic analysis and possibly other alternatives to soldering, such as one piece casting and laser welding.

# APPENDIX

LIST OF MATERIALS AND MANUFACTURERS:

Dentsply International
550 West College Avenue
P.O. Box 872
York, Pennsylvania 17404
Biobond alloy
Biobond solder #6-10-CB-Strip
Biobond Soldering Flux

Howmedica, Inc.
5101 South Keeler Avenue
Chicago, Illinois 60632
Microbond $NP_2$ Batch #762335
Microbond NP Strip Solder #06-058
Microbond NP - Post-Solder #042180
Microbond Pre-Solderin Flux #060037
Microbond Post-Soldering Flux #040044

Instron Corporation
2500 Washington Street
Canton, Massachuettes 02021
Instron Model #1125

Ceramco, Inc.
20 Lake Drive
E. Windsor, New Jersey 08520
Ceramalloy II alloy
Ceramalloy NP solder #00001
Ceramalloy Soldering Flux #0E002

Maves Company
P.O. Box 44004
Cleveland, Ohio 44144
Mave's Inlay Wax (C240 3T)

Perkeo Company
West Germany
Perkeo 2-4 - Tip
Perkeo Multiplex - Torch

Teledyne Dental Products
Elk Grove Village, Illinois 60007
Vescote lot #801-001-02

Unitek Corporation
2724 S. Peck Road
Monrovia, California 91016
Unibond Alloy #31280-7
Unibond Non-Precious Solder

C. Vaupel Dental Inc.
10804 Fallstone Road - Suite 210
Houston, Texas 77099
Cospan
Cospan Solder
Cospan Soldering Flux

Whip-Mix Corporation
361 Farmington Ave.
Louisville, Kentucky 40217
Hi-Temp Investment lot#0176101
High-Heat Investment lot#0980001

Williams Gold Refining Company
2978 Main Street
Buffalo, New York 14214
Williams Bondal Flux
Litecast B alloy lot#43501 9B
"Super solder" #23773M

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# VITA

William Gregory Kaylakie was born in Atlantic City, New Jersey on September 6, 1951; son of John E. Kaylakie and Mary Croener Kaylakie. He graduated from Atlantic City High School in 1969. That same year he enrolled at Fairleigh Dickinson University, Teaneck, New Jersey; from which he was awarded, cum laude, a B.S. degree in Biology in 1973. He attended Fairleigh Dickinson University School of Dentistry from 1972 to 1976, from which he received his D.M.D. degree. During this time, he married Marcia Roberts on December 28, 1974.

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This thesis is submitted for partial fulfillment of a Master of Science degree to be awarded upon completion of requirements, December 1981, from the University of Texas Health Science Center at San Antonio School of Biomedical Sciences.